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ENVIRONMENTAL EFFECTS OF A STEARATE COATING ON THE FRACTURE BEHAVIOR OF GUN STEEL

July 1976

BENET WEAPONS LABORATORY
WATERVLIET ARSENAL
WATERVLIET, N.Y. 12189

TECHNICAL REPORT

AMCMS No. 07001

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A stearate coating has been proposed for lead as a lubricant in the swage autofrettage process for cannon tubes. In this study, the environmental effects of this coating and the various chemical compounds used in its formation on the fracture behavior of gun steel have been evaluated at various temperatures up to 675°F. No environmental embrittlement effects were observed on crack initiation in smooth specimens tested in tension monotonically or in constant load delayed failure type tests. Similarly, minimal effects were observed on crack propa-

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gation in single edge notched flat bar specimens which were tested to failure in fatigue. These results are in agreement with the scanning electron microscope examination of the fracture surface of the specimens. Thus, from the standpoint of environmental embrittlement, the stearate coating is superior to lead as a lubricant in the swage autofrettage process.

X-ray diffraction studies of the stearate coating revealed that it consists of β-sodium stearate rather than zinc stearate as reported in the literature. β-sodium stearate does not react with gun steel and forms a loosely adhering surface coating as compared to a good intimate coating formed with lead. Thus, the use of stearate coating as an effective lubricant may have some limitations in the swage autofrettage processing for cannon tubes.

WVT-TR-76029

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ENVIRONMENTAL EFFECTS OF A STEARATE COATING ON THE FRACTURE BEHAVIOR OF GUN STEEL

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I. INTRODUCTION

Lead electroplated inside the bore of a gun tube has historically been used as a lubricant for the mechanical swage autofrettage process. After the post-autofrettage thermal treatment at 675°F for four hours, occasional transverse cracks were observed. In instances, these cracks were around the entire circumference and nearly through the wall thickness and resulted in complete fracture during subsequent gun tube straightening[1] (figure 1). Recent study has shown that this cracking phenomena is due to liquid metal embrittlement; the liquid lead acting as embrittling species in combination with high longitudinal tensile residual stresses present in the bore surface of the cannon.

A recently developed stearate coating process[2] offers a potential substitute for lead as a swage autofrettage process lubricant. The purpose of this study is to evaluate this proposed coating in terms of its environmental embrittlement behavior under the conditions of the swage autofrettage-post thermal treatment process. The effects of the coating itself and the chemical compounds used in forming the coating are examined with regard to crack initiation and cyclic crack growth characteristics of gun steel at certain characteristic temperatures, specifically at the temperature of the post-autofrettage thermal treatment. In addition, x-ray diffraction studies

1. P. Thorton, Transverse Cracking in 105mm M68 Forgings", Interim Progress Report, Watervliet Arsenal, 1975.
2. H. Goodheim, "Engineering Study and Evaluation of Zinc Stearate Lubricant for Swage Autofrettage", Technical Report, Watervliet Arsenal, July 1974.

were undertaken to determine the various compounds formed during the coating process and to identify the specific compound(s) that act as a lubricant.

II. EXPERIMENTAL

(a) Crack Initiation Studies: Smooth tensile specimens (.505" in diameter and with 2.5" gauge length) were machined from the material near the bore surface of a gun tube. These were coated with the stearate lubricant using the chemicals and the process described in Ref. 2 and given below. The specimen was cleaned first in acetone and subsequently etched electrolytically in potassium hydroxide-potassium carbonate solution for thirty seconds at nine volts and then rinsed in hot water. Then it was immersed for ten minutes in a five percent solution of Granodraw No. 72 (zinc phosphate) at 185°F and subsequently rinsed in hot water. After this treatment, the specimen was immersed in a sodium stearate solution (.85#/gal) for five minutes at 185°F and was dried for several minutes. Subsequently it was air dried, had a white stearate coating, and was ready to test. Thicker coatings of the lubricant were obtained by increasing the processing times in various chemical baths. The tests were performed in the Rhiele tensile testing machine. An environment chamber made of a steel tube (2" dia. and 8" high) was welded to the loading rods and provided a reservoir for the specific environment in which the specimens were tested. A

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2. H. Goodheim, "Engineering Study and Evaluation of Zinc Stearate Lubricant for Swage Autofrettage", Technical Report, Watervliet Arsenal, July 1974.

tube furnace enclosed the tensile specimens and the environment chamber and was used to test specimens at elevated temperature in flowing argon atmosphere. The top opening of the furnace was sealed with an iron plate and a high temperature cement. An inlet for argon in the sealed iron plate allowed the specimens to be tested in a flowing argon atmosphere. The bottom opening of the tube furnace was sealed with glass wool and asbestos, etc. The environments used were argon, zinc stearate, sodium stearate, zinc phosphate (10% solution by vol. of zinc in phosphoric acid, i.e. Grandodraw No. 72-Alchem Products, Ambler, Pa) and the stearate coating environment produced on the surface of the steel (i.e. using the stearate coating process described earlier, fine (200 mesh) iron particles were coated with the stearate coating and this stearate coated iron powder was used as an environment). The sodium and zinc stearate compounds are solids at room temperature. These environments, therefore, were either packed around the coated tensile samples or were melted and poured in the environment chamber. These environments melt, evaporate and perhaps decompose when tested at elevated temperatures. Therefore, sufficient environment was provided in the environment chamber to ensure the presence of the environment during the test when specimens were tested at various temperatures*, and at 675°F, the temperature of the post-autofrettage thermal treatment.

* The specimens were tested at 185°F in the zinc phosphate solution, since this is the temperature used in the coating process. Zinc stearate is liquid at 315°F and, therefore, in this environment the specimens were tested at 315°F, as well as 675°F, the temperature of the post-autofrettage treatment. Sodium stearate does not appear to have a definite liquidification temperature and therefore specimens in sodium stearate were tested at 675°F.

The specimens were loaded at room temperature just past the yield stress and prestrained a few percent and this load was maintained constant while the specimens were heated to desired temperatures. When at temperature, the specimens were loaded in increments of 250 to 500 lb and were tested in tension to failure in as-coated condition and in as-coated condition in various environments. The yield and the fracture stress, UTS and reduction in area and percent elongation at failure, etc. were obtained from the load-extension curve and the fractured specimen. In all tests performed with specimens contained in the environment chamber, only fracture stress UTS and reduction in area at fracture were obtained. The load-extension curves were not obtained because an extensometer could not be clamped to the specimen when the environment chamber was used. The fracture surfaces of the specimen were examined by scanning electron microscopy for cracks or the mode of failure (i.e. ductile or brittle), etc.

(b) Crack Propagation Studies: Crack propagation studies, were carried out with single edge notched, flat tensile specimen shown in Figure 2.[3] The specimens were first compression loaded to 2700 lbs. Subsequently, these were mounted in a fatigue testing machine and a fatigue crack was produced at room temperature at 1800 rpm using a tensile load of 1950 lbs. The number of cycles to produce the crack and its approximate crack length was measured. The precracked specimens were then coated with lubricant using the procedure described

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3. W. F. Brown and J. W. Strawley, ASTM Special Technical Publication No. 410", Plain Strain Crack Toughness Testing of High Strength Metallic Materials, 1960.

earlier. The specimens were tested in a Sontag fatigue testing machine and were enclosed by an environment chamber which was welded to the loading rod as described earlier in section II (a). The environment chamber was filled with desired environment as described earlier. A split furnace enclosed the specimen, the environment chamber and the inlet for argon. Glass wool was packed around the loading fixtures and the open ends of the furnace. The specimen was heated to 675°F in flowing argon atmosphere. A chromel-alumel thermocouple was inserted in the environment and the temperature was measured using a potentiometer. The specimen was then loaded to 1950 lbs. in tension and fatigue tested to failure at 1800 rpm. The number of cycles to failure was recorded. During testing, the environment, particularly zinc and sodium stearate, gave out considerable amounts of fumes and gases and black decomposition products coated the fracture surface of the specimen. This coating on the fracture surface was removed by chemical treatment, nevertheless parts of crack front and the fracture surface remained contaminated with test environment and black decomposition products. Subsequent to testing, the crack length was measured. The measured crack lengths are considered very good but not extremely accurate. The fracture surface was examined for cracks at or in the vicinity of the fatigue crack front and elsewhere on the fracture surface. The embrittlement or its absence in the environment was determined by comparing the fracture mode or characteristic features (i.e. intergranular, transgranular or ductile failure) of the fracture surface observed using a scanning electron microscope, the stress intensity, crack length, and

the number of cycles to failure in fatigue in the environment with that in argon, the inert environment.

(c) X-Ray Diffraction Studies:

X-ray diffraction studies were performed on six round tensile specimens coated with the stearate process described earlier to identify the chemical compounds present in various layers starting from the steel surface. Three of the coated specimens were supplied as typical of the production stearate coating process. X-ray diffraction patterns from pure chemical compounds were also obtained to serve as standards e.g. zinc stearate, etc. These are the chemicals used in the coating process and were anticipated to be present in the coating.

III. RESULTS

The mechanical test data for crack initiation in smooth tensile specimens and single edge-notched flat tensile bars tested in fatigue in coated condition, in coated condition with various environments and for comparison in argon in the uncoated condition are given in Tables I and II. The data represents reproducible results of two to six or more tests.

The smooth specimens used for crack initiation studies in various environments and a test temperature of 675°F, exhibited the same yield stress, ultimate tensile stress, the fracture stress, reduction in area as those for specimens tested in inert argon atmosphere under identical conditions, Table I.

The test data on single edge-notched flat precracked bars, tested in fatigue to failure given in Table II show the number of cycles to

failure and the critical crack length, a. The critical stress intensity at the root of the fatigue crack at the point of failure, (K), was calculated using the crack length, a, the width of specimen b, the applied tensile stress σ, the equation $K = \sigma \sqrt{\pi a}$ (F(a/b), and the calibration curve given in Figure 3. This calibration curve and the equation for deriving K values for single edge-notched flat precracked specimens tested in tension were taken from ASTM publication No. 410[3]. The data in Table II show that the stress intensity and cycles to failure are essentially independent of the environments and test temperatures used.

Scanning electron micrographs of fracture surfaces of smooth and fatigue tested specimens are shown in Figures 4 thru 7 and 8 - 21 respectively.

The results of the x-ray diffraction studies established that the dark brown coating closest to the steel surface for all the six stearate coated rods was zinc ortho-phosphate hydrate (Hapetite) $Zn_2(PO4)_2$ 4.H_20. The white coating immediately adjacent to this layer as well as the outer most white coating was identified as β-Sodium stearate, $C_{17}H_{35}$ Coona. Zinc stearate was not found in any of the layers in the coatings.

IV. DISCUSSION

The results of the mechanical test data on crack initiation in gun steel in the presence of the stearate coating and in all chemical

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3. W. F. Brown and J. W. Strawley, ASTM Special Technical Publication No. 410", Plain Strain Crack Toughness Testing of High Strength Metallic Materials, 1960.

environments used during the application of the stearate lubricant to the bore surface of gun tubes and at all test temperatures including 675°F, the post-autofrettage thermal treat are effectively identical to that for crack initiation in inert argon atmosphere. Also, the results show that no degradation in properties occurred in zinc phosphate solution at 185°F, the temperature used in processing the steel and also at 315°F, the temperature at which zinc stearate is liquid. The fracture mode is ductile at all test temperatures in all environments and is identical to that in inert argon atmosphere. The fracture in all cases was of the cup-cone type. Scanning micrographic examination revealed a dimpled type of fracture with no dependency upon environment as seen by comparing Figures 4-5 with 6-7. The scanning macro and micrographs of specimens tested in sodium stearate and other environments were similar to those for zinc stearate and therefore are not included here. Based on above, it is concluded, that the stearate coating itself and various other environments used in the application of the stearate coating have no embrittling effects on crack initiation in gun steel.

Most failures in metals are propagation rather than nucleation controlled[4]. Therefore, crack propagation tests performed with single edge notched pre-cracked tensile bars constitute a severe test for evaluating the embrittling effects of active chemical environments. The results of fatigue tests given in Table II show that the value of

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4. M. H. Kamdar, "Embrittlement by Liquid Metals", Prog Mat. Sci., Vol. 15, No. 4, 1973, Pergamon Press, Ed. Chalmers, et al.

critical stress intensities range from 91 to 106 ksi $\sqrt{in}$. The number of cycles from the initial pre-crack to failure in argon, zinc and sodium stearate at 675°F are about the same, i. e. 25,000 - 32,000 cycles and so are the critical crack lengths, approximately one inch long. The values of critical stress intensities reported however, may not represent a valid value for gun steel at 675°F, because the critical lengths are very long (~1") and approach the width of the specimen (1.25"). Under these conditions, the values reported here may not meet the ASTM requirements for a valid test. Nevertheless, for embrittlement studies, it is the relative rather than the absolute values of critical stress intensity that are of concern. For this purpose the test results are adequate.

The results cited above show that the fatigue and fracture behavior of gun steel is not effected by any of the environments of concern and the propensity towards any embrittlement is minimal. This is further borne out by the chracteristic features of the fracture surfaces, i. e. whether cracking is brittle or ductile. Figures 8 thru 13 show the scanning electron micrographs of the fracture surface of specimens tested in argon at 675°F at the crack front and at various regions away from it. It is seen that fracture is predominantly ductile with some intergranular cracks. Similarly, Figures 14 thru 21 show ductile failure with intergranular cracks for specimen tested in sodium and zinc stearate. A comparison of the Figures 8 through 21 shows that the characteristic of the fracture surfaces in both argon and stearate environments are essentially identical. A possibility of embrittlement exists near the edge of the specimen tested in stearate where

intergranular cracks were observed, Fig. 21. These cracks may be caused by hydrogen or methane gases which are known to embrittle steel. These gases may be produced by degradation of stearate heated to 675°F. However, cracking in these environments is slow crack growth controlled and will not be as catastrophic and severe as that observed in liquid lead environment.

The results on crack initiation and fatigue crack propagation on gun steel and fractographic observations in various environments at 675°F suggest that during post autofrettage thermal treatment the possibility of environmental embrittlement is minimal. Therefore, the stearate lubricant proposed for swage autofrettage is superior to lead as far as severe embrittlement is concerned. The stearate coating, therefore, could be used with minimum concern about chemical embrittling effects.

The x-ray diffraction results are interesting. These results clearly indicate that the coating consists of β-sodium stearate only and zinc stearate is absent in the coatings. Thus, β-sodium stearate is the lubricant rather than zinc stearate as has been reported in the literature. Other chemical studies performed indicate that zinc phosphate does not appear to react with sodium stearate to produce zinc stearate on the steel surface. The absence of such a chemical reaction and the results from x-ray diffraction studies indicate that the lubricant β-sodium stearate is loosely adhering to the zinc phosphate etched surface of the steel. Since the coating is loosely adherent, care should be exercised to insure its uniformity and lack of flaking in order to perform satisfactorily as a swage autofrettage lubricant.

V. CONCLUSIONS

(1) Various chemical environments used in stearate coating process have no embrittling effects on crack initiation in gun steel at 675°F, the post swage autofrettage thermal treatment temperature.

(2) These environments have minimal if any embrittling effects on crack propagation at 675°F. In this regard stearate coating is superior to lead.

(3) X-ray diffraction studies have established that the coating is β-sodium stearate and not zinc stearate as reported in the literature. Also chemical reaction does not seem to occur between zinc phosphate and sodium stearate to produce the zinc stearate. The β-sodium stearate coating is flacky and adheres loosely to the etched zinc phosphate coated surface of steel.

VI. REFERENCES

1. P. Thorton, Transverse Cracking in 105mm M68 Forgings", Interim Progress Report, Watervliet Arsenal, 1975.
2. H. Goodheim, "Engineering Study and Evaluation of Zinc Stearate Lubricant for Swage Autofrettage", Technical Report, Watervliet Arsenal, July 1974.
3. W. F. Brown and J. W. Strawley, ASTM Special Technical Publication No. 410", Plain Strain Crack Toughness Testing of High Strength Metallic Materials, 1960.
4. M. H. Kamdar, "Embrittlement by Liquid Metals", Prog Mat. Sci., Vol 15, No. 4, 1973, Pergamon Press, Ed. Chalmers, et al.

TABLE I

Test data for smooth 0,505" dia, tensile specimens tested to failure in stearate coated (c) condition in various environments.

Environment	Temperature	Yield Stress ksi	Ultimate Tensile Stress ksi	Reduction in area at failure,%	Failure	Comments
Coated (c)	675°F	135	160	60	Ductile	
Argon (un-coated)	675°F	138	160	55	Ductile	
C+ Zinc stearate	315°F	-	180	25	Ductile	Specimen had 0.25" hole thru center of gage
C+ Zinc stearate	675°F	135	157	66	Ductile	
C+ Sodium stearate	675°F	135	160	60	Ductile	
C+ Zinc phosphate solution	185°F	150	180	46	Ductile	
C+ Zinc phosphate solution	675°F	135	170	60	Ductile	
C+ Coated fine iron powder	675°F	135	160	62	Ductile	

TABLE II

Flat notched bars fatigue precracked at room temperature at 1800 rpm and 1950 lbs. The bars were coated with stearate coating and fatigue tested to failure at 1800 rpm and 1950 lbs in tension in various environments.

Environment	Temp.	Critical Crack Length (Inches)	Critical Stress Intensity, K, ksi $\sqrt{in}$	Cycles to failure, (10^3)
Argon (un-coated)	675°F	1.00"	105.5	29
As coated	675°F	1.00"	105.5	32
Zinc stearate	675°F	0.98"	91.1	31
Sodium stearate	675°F	0.98"	91.1	32
Zinc Phosphate solution	185°F	1.01"	106.0	25

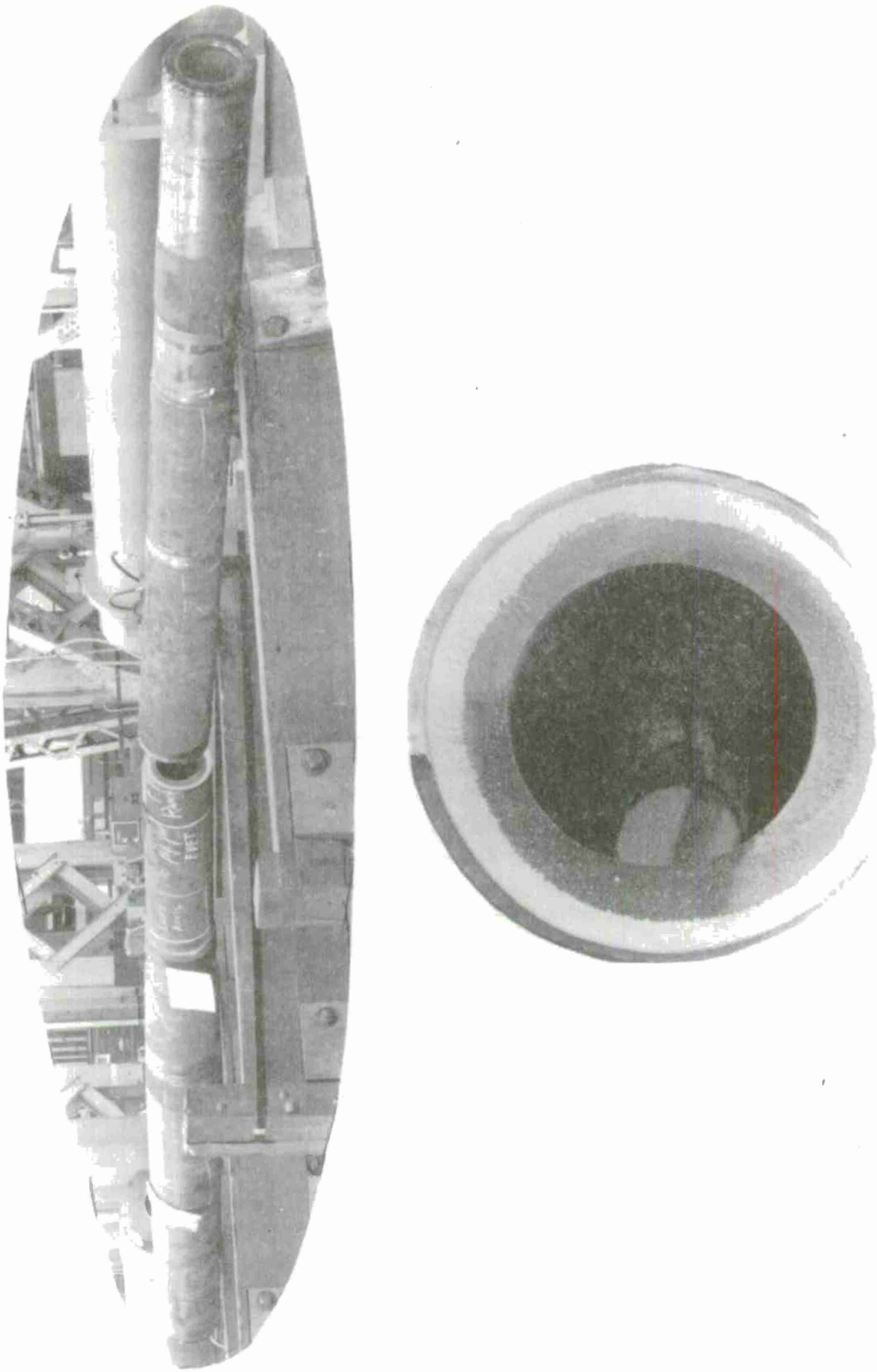

Fig. 1. Lead coated gun tube which broke brittlely in two pieces after heat treatment at 675°F for 4 hrs.

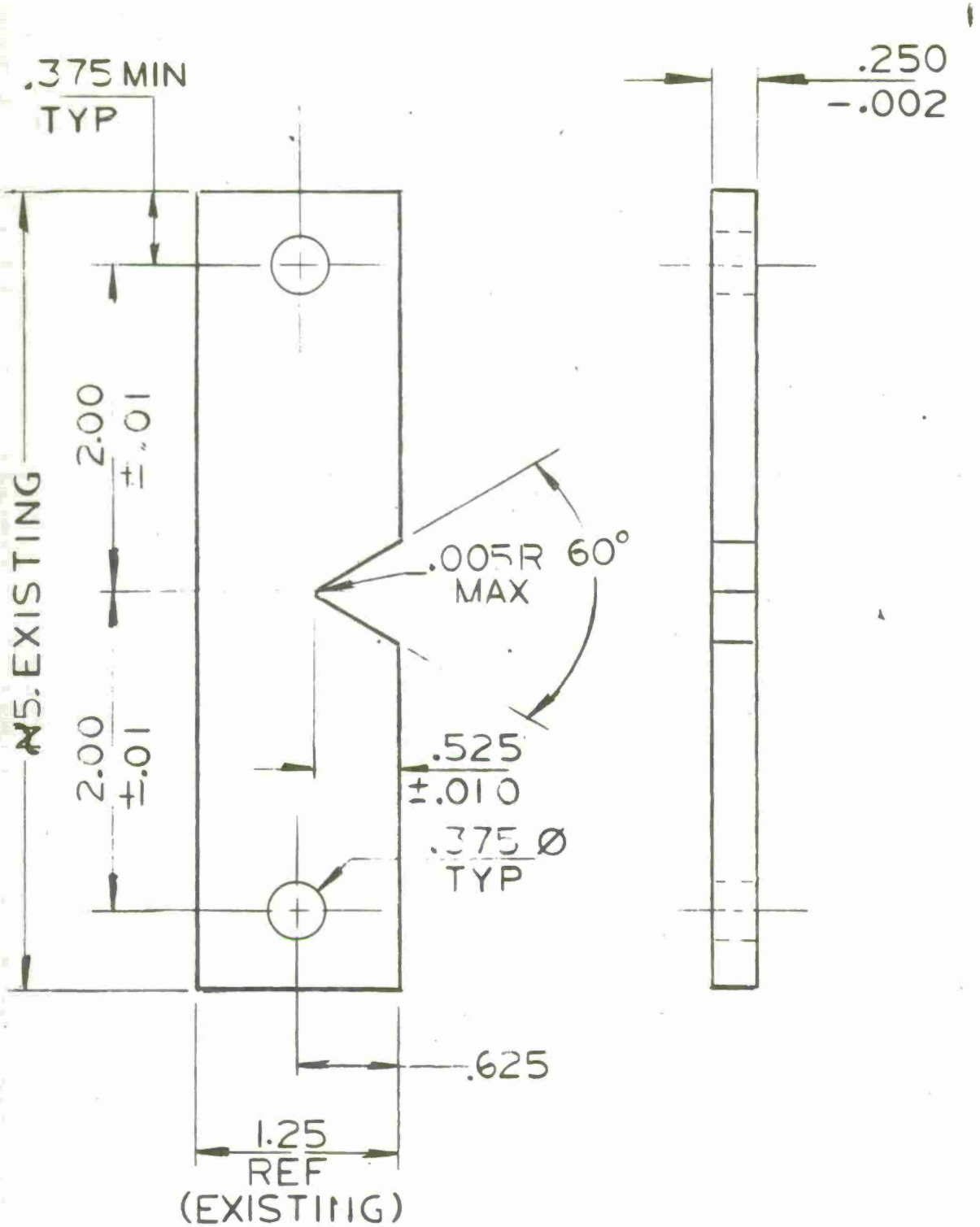

Fig. 2

SINGLE EDGE NOTCHED FATIGUE TENSION TEST SPECIMEN

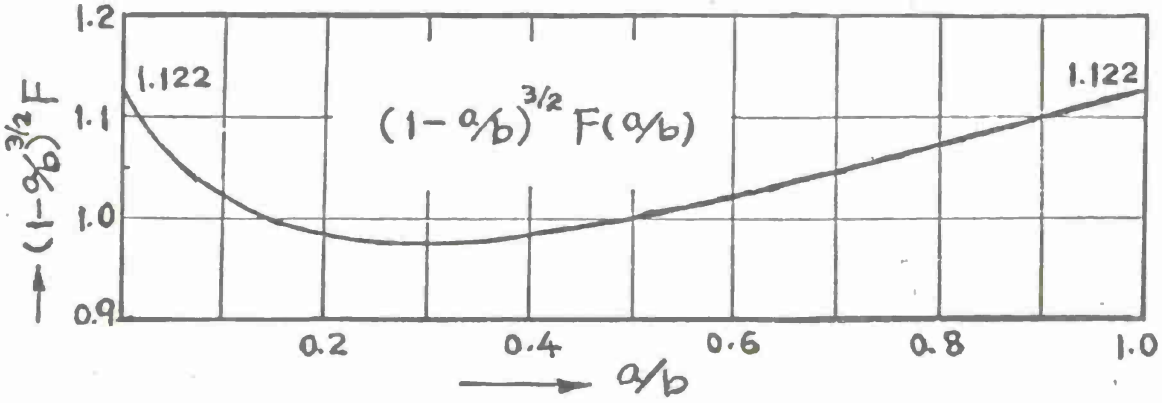

Fig. 3. CALIBRATION CURVE FOR SINGLE EDGE NOTCHED TENSION FATIGUE TEST SPECIMEN

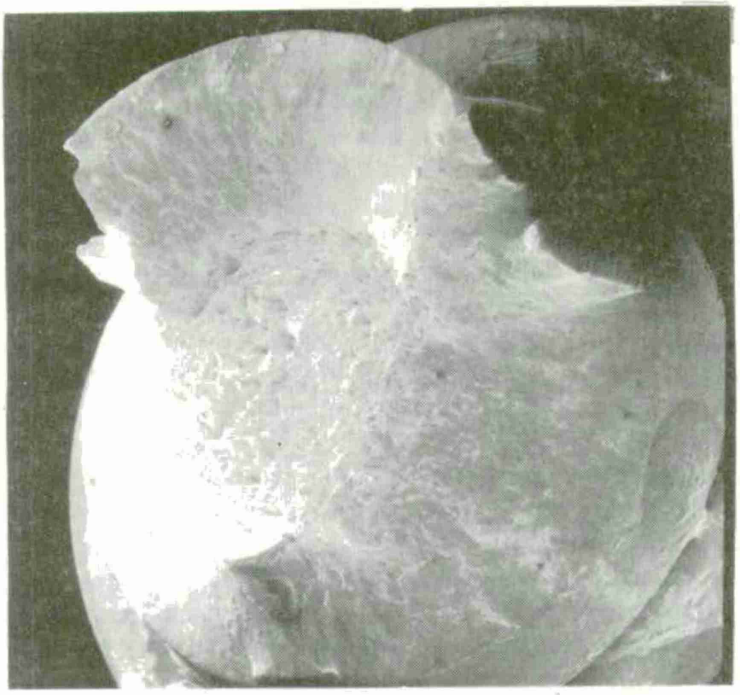

Fig. 4. Ductile failure of smooth specimen tested in tension at 675°F in argon, mag 10X

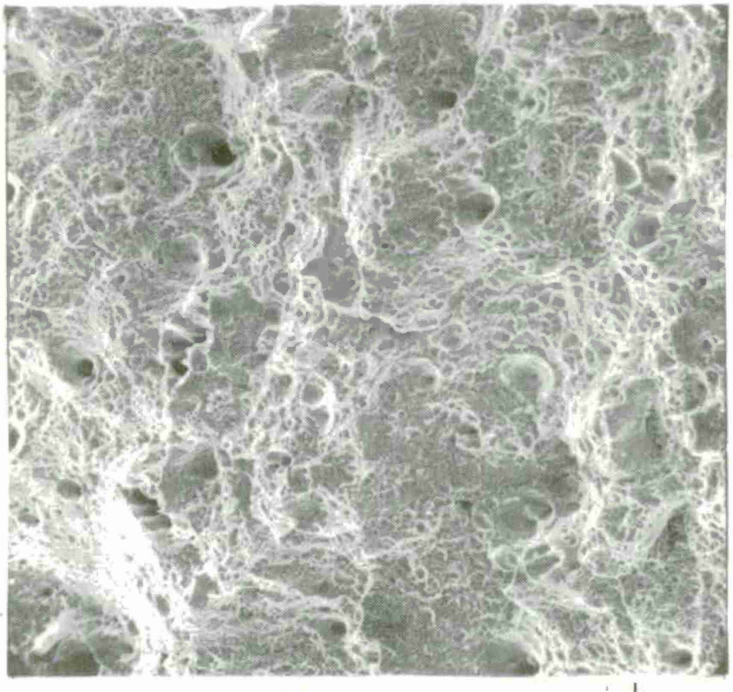

Fig. 5. A high magnification micrograph (mag440X), showing ductile failure of the fracture of specimen in Fig. 4.

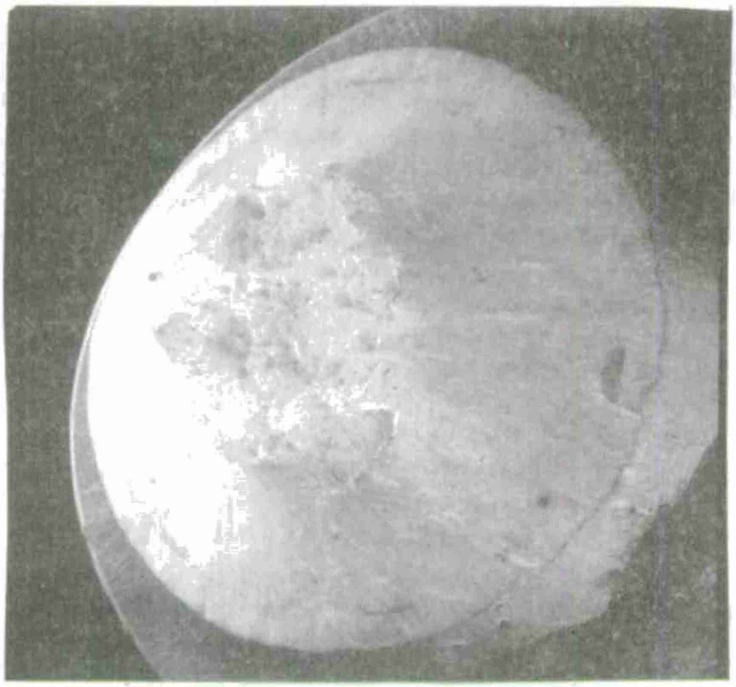

Fig. 6. Ductile failure of smooth specimen tested in tension at 675°F in zinc stearate environment, mag 10X.

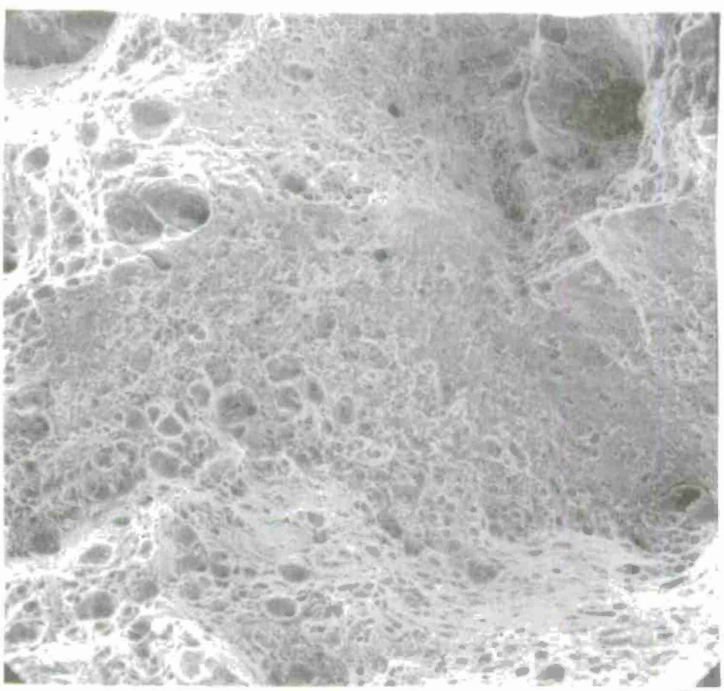

Fig. 7. A high magnification micrograph (mag 440X) showing ductile failure in specimen shown in Fig. 6.

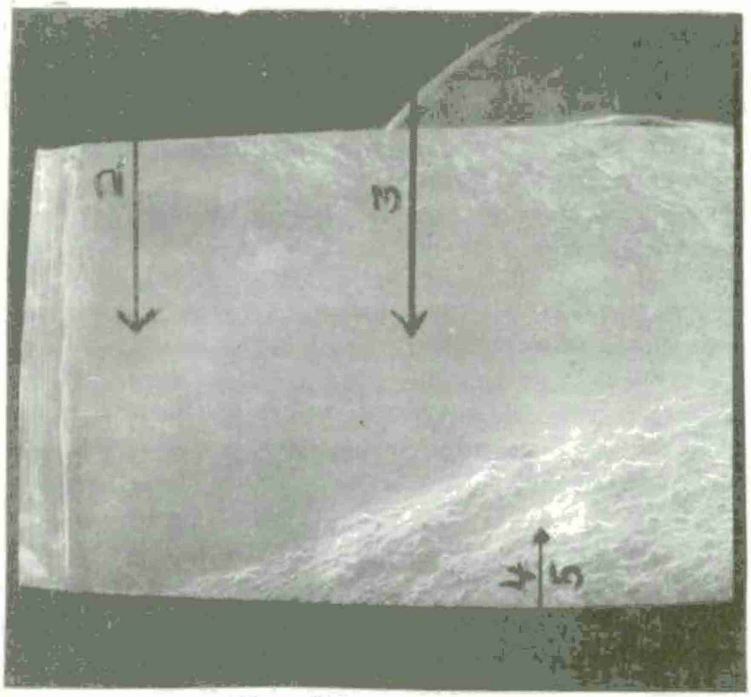

Fig. 8

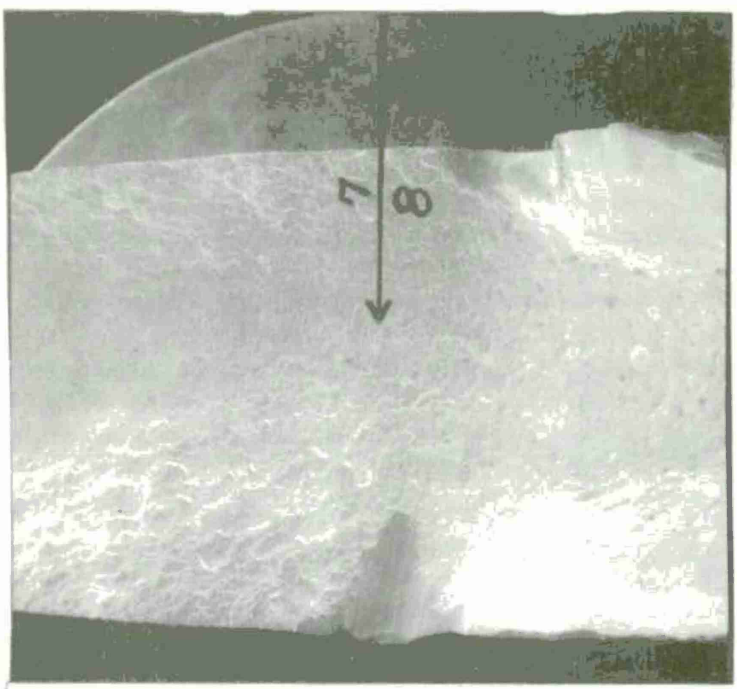

Fig. 9

Figs. 8 & 9. Show regions at crack front and away from the crack front for a specimen tested in argon at 675°. which were examined at higher magnifications.

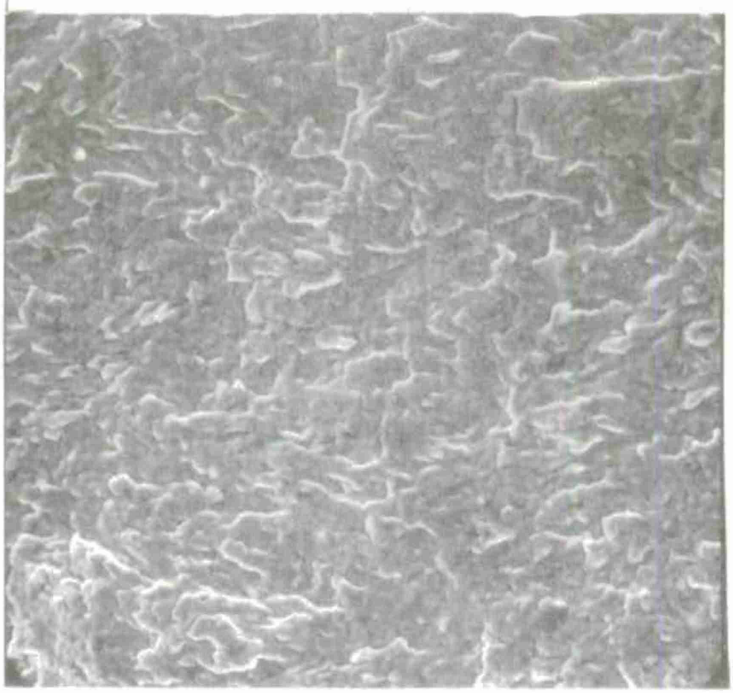

Fig. 10

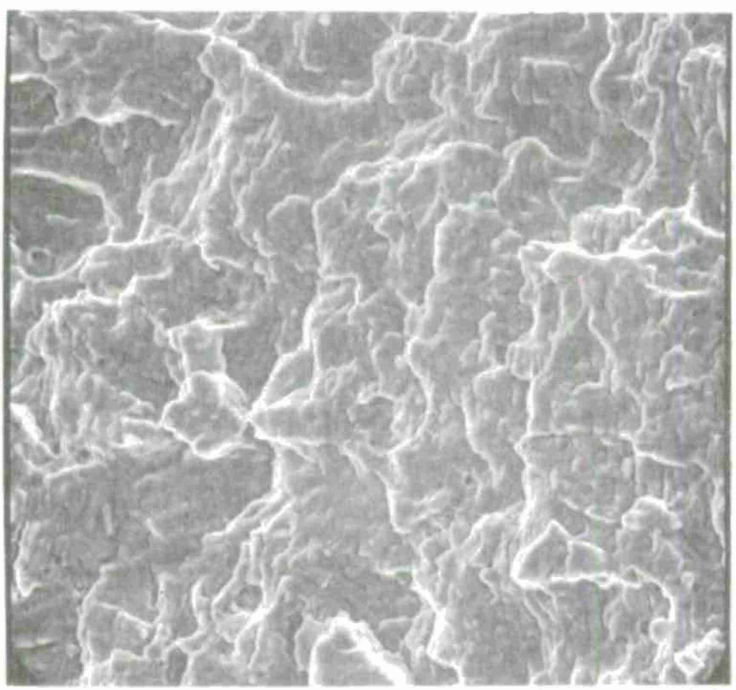

Fig. 11

Figs. 10 & 11. These micrographs show that in regions 2 and 3 on the fracture surface in Fig. 8, are essentially ductile, Mag 960X.

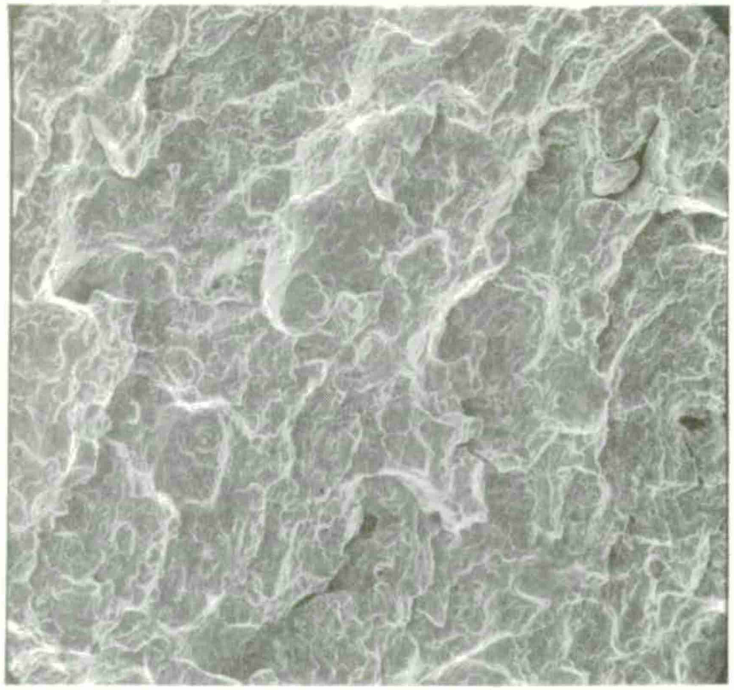

Fig. 12

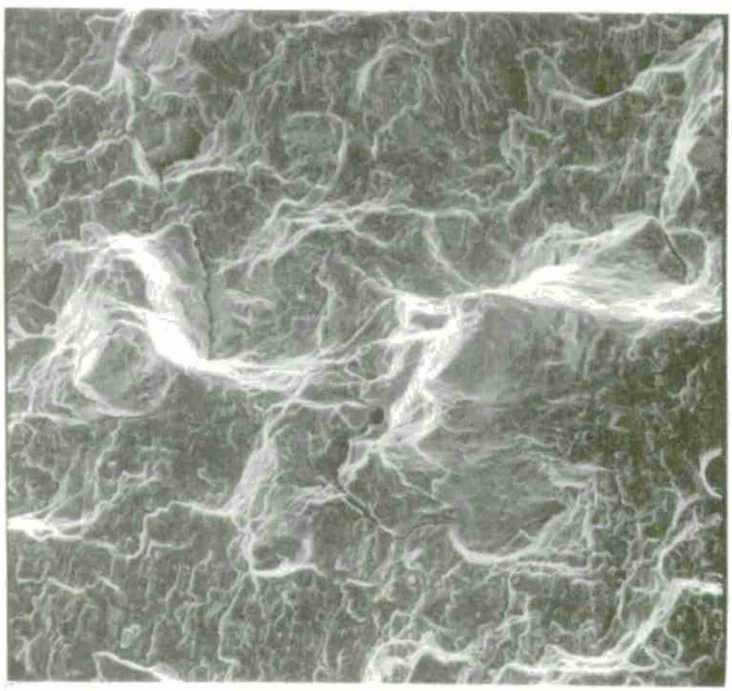

Fig. 13

Figs. 12 & 13. These micrographs show intergranular cracks in region 7 and 8 in Fig. 9.

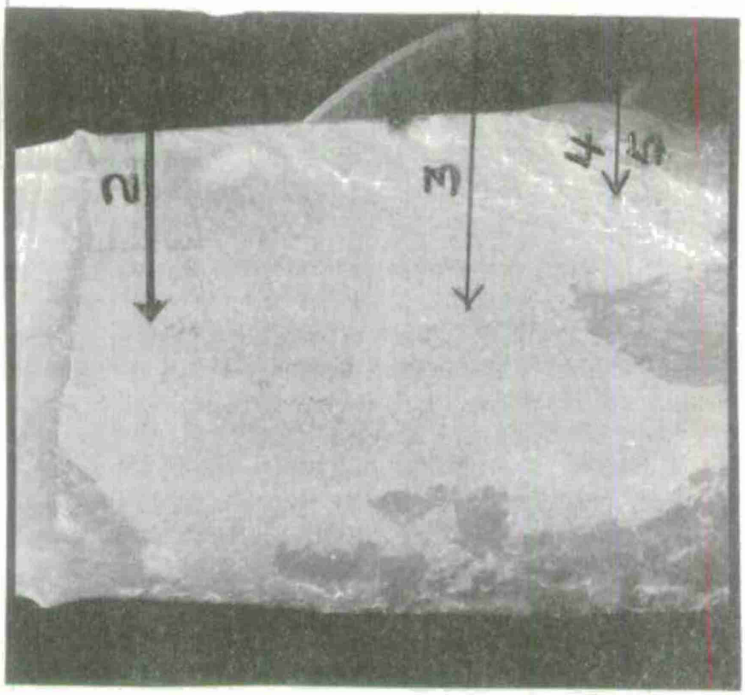

Fig. 14. Fracture surface showing various regions which were examined at high magnification for a specimen tested in fatigue at 675°F in sodium stearate.

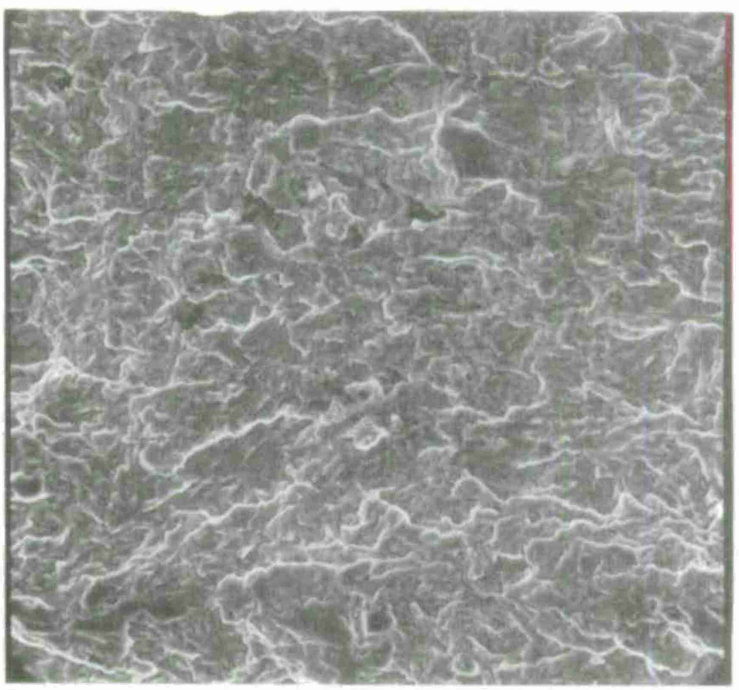

Fig. 15. Shows ductile failure in region 2, in Fig.14, near the fatigue crack front, mag 960X.

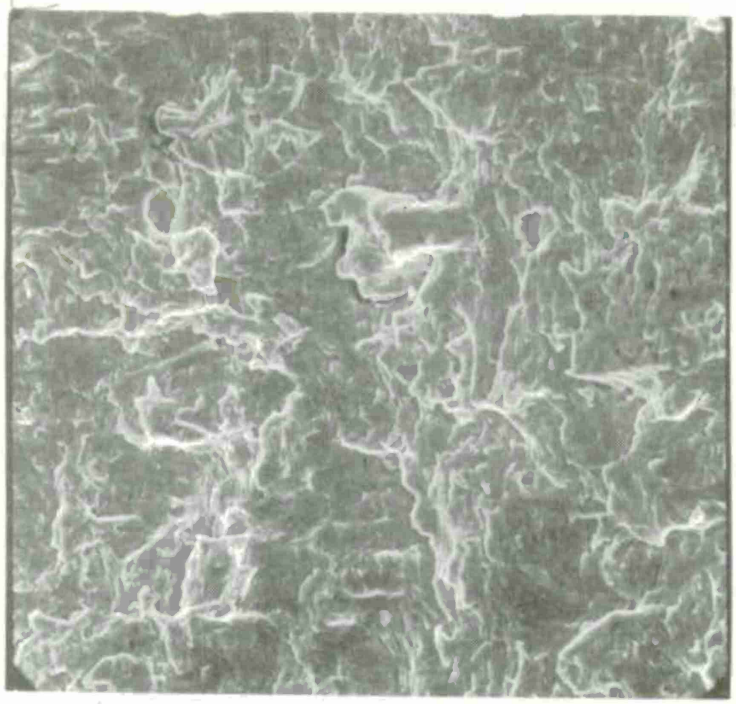

Fig. 16

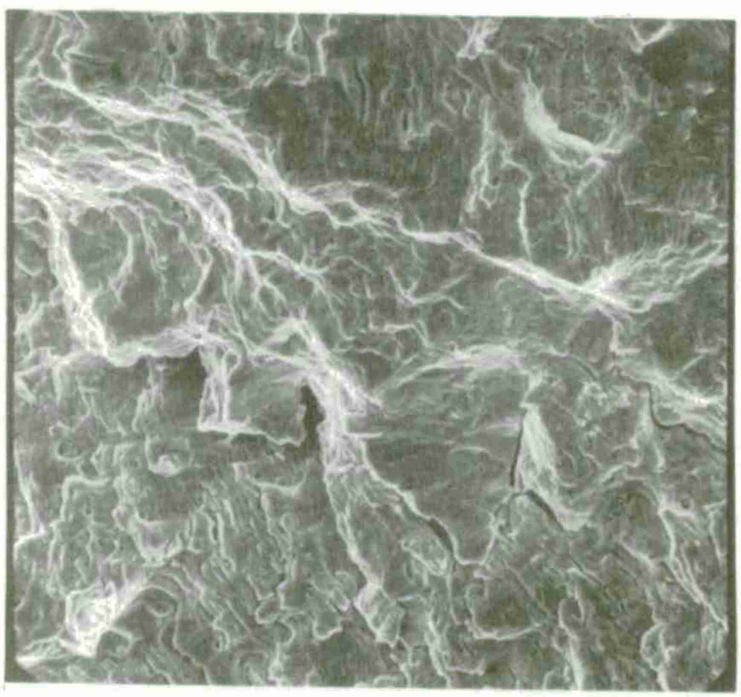

Fig. 17

Figs. 16 & 17. Show the fracture surface in regions 3 and 5 in Fig. 14. Notice that fracture is mixed, i.e. ductile with intergranular cracks mag 960X.

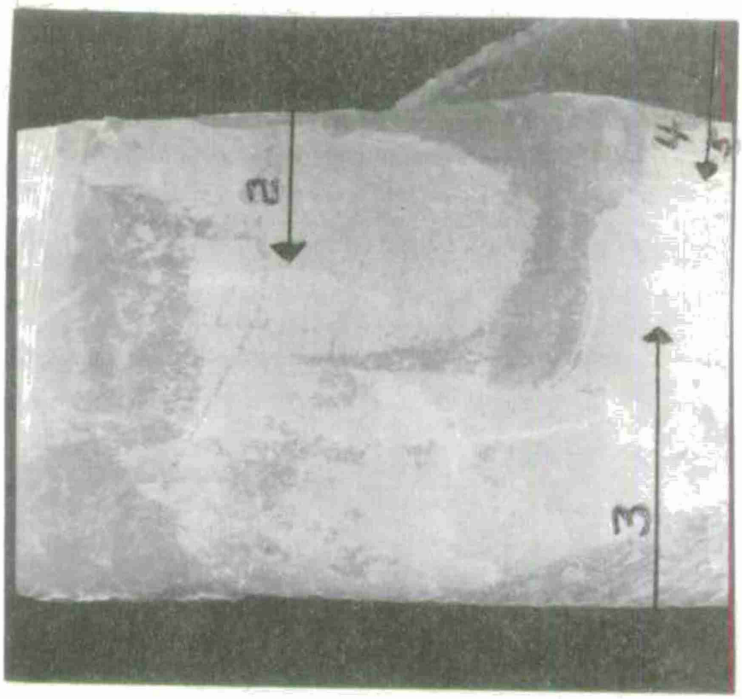

Fig. 18. Fracture surface showing various regions which were examined at highest magnification for a specimen tested in fatigue in zinc stearate at 675°F.

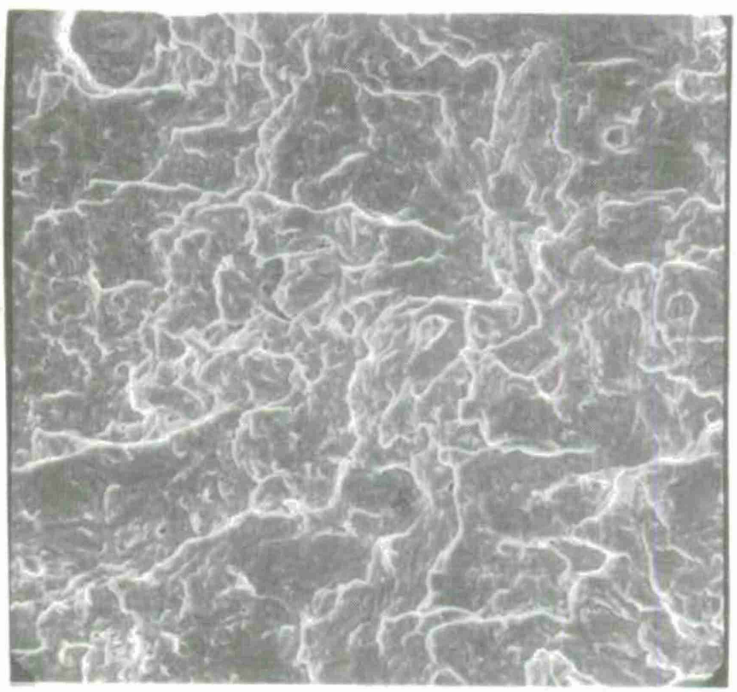

Fig. 19. This figure shows essentially ductile failure in region 2 near the fatigue crack front, mag 960X.

Fig. 20. Shows the fracture surface appearance in region 3 which is similar to that in Fig. 19, 960X.

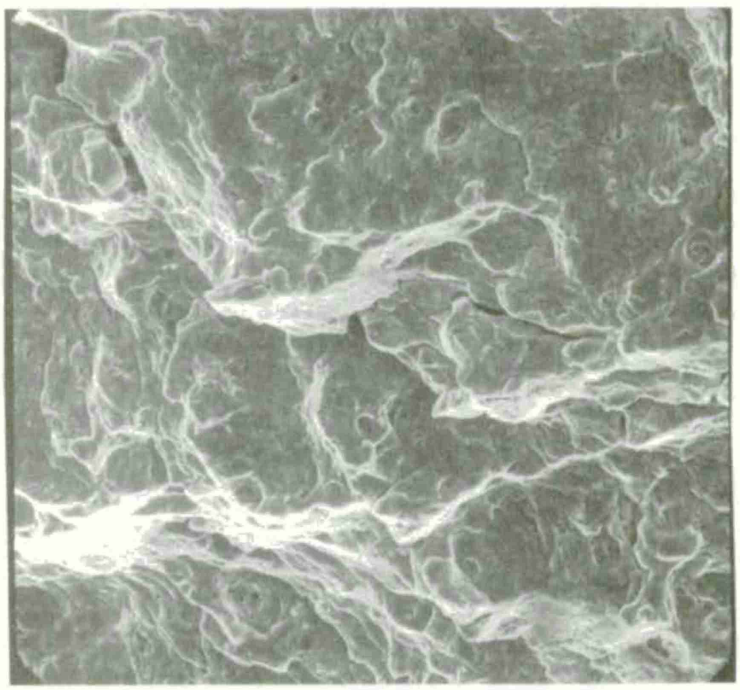

Fig. 21. The fracture surface in region 5, showing mixed ductile and intergranular failure, mag 960X.

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